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*Title*

THE PREDICTION OF  
OXIDATION RATES OF CARBONACEOUS  
MATERIALS FROM PLASMA ARC TESTS

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by

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## ABSTRACT

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A series of stagnation point plasma arc tests on specimens of carbonaceous material was examined to find out whether the loss of mass could be explained in terms of oxidation of the carbon. Up to a surface temperature of 4000°F, it was found that it could be predicted in terms of the rate of diffusion of oxygen through the boundary layer, computed by Lewis analogy from the known heat flux and driving enthalpy. Best results were obtained by assuming that carbon monoxide was formed, and by using the net heat flux corrected for hot wall and transpiration cooling effects. One series of specimens exhibited marked oxidation resistance, and here the loss rate conformed to the half order Arrhenius equation, in which Lewis analogy was again used to compute the partial pressure of oxygen near the wall. In this case it was possible to compute the reaction rate coefficient of oxidation for the material. Above 4000°F, it was found that additional mass loss was caused by spalling. This was a strong function of temperature, and could be correlated by an equation of the Arrhenius form, although insufficient data was available to determine pressure effects. It is concluded that sufficient information can be obtained from plasma tests to compute the oxidation and spalling rates of a carbonaceous material on a re-entry vehicle.

AUTHOR

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# SYMBOLS

- A = Reduced reactivity,  $\text{lb/ft}^2\text{-sec-atm}^{1/2}$
- B = Diffusion parameter, dimensionless
- C = Spalling constant,  $\text{lb/ft}^2\text{-sec}$
- $C_{ox}$  = Mass fraction of oxygen
- $c_p$  = Specific heat of fluid,  $\text{BTU/lb}^\circ\text{R}$
- D = Diffusion coefficient for oxygen,  $\text{ft}^2/\text{sec}$
- $D = K_o e^{-T_{ox}/T_w}$ , dimensionless
- E = Activation energy of oxidation, K cal/mole
- $h_{eff}$  = Effective skin coefficient,  $\text{LB/ft}^2\text{-sec}^\circ\text{R}$
- $i$  = Enthalpy of fluid,  $\text{BTU/lb}$
- $K_o$  = Dimensionless reaction rate coefficient
- $K_s$  = Dimensionless spalling coefficient
- $k$  = Conductivity of fluid,  $\text{BTU/ft-sec}^\circ\text{R}$
- $k_{ox}$  = Reaction rate coefficient of oxidation,  $\text{lb/ft}^2\text{-sec-atm}^{1/2}$
- $L = e D c_p / k$
- $L_{eff}$  = Effective Lewis number
- $\dot{M}$  = Mass loss rate,  $\text{lb/ft}^2\text{-sec}$
- $\dot{M}_e$  = Rate of loss due to oxidation,  $\text{lb/ft}^2\text{-sec}$
- $\dot{M}_{ox}$  = Rate of diffusion of oxygen,  $\text{lb/ft}^2\text{-sec}$
- $\dot{M}_{sp}$  = Rate of loss due to spalling,  $\text{lb/ft}^2\text{-sec}$
- $\dot{M}_{tot}$  = Total rate of loss,  $\text{lb/ft}^2\text{-sec}$
- $P_{ox}$  = Quantity used in determining oxidation constants
- $P_{spall}$  = Quantity used in determining spalling constants
- $p$  = Local static pressure, atmospheres

$P_{O_2}$  = Partial pressure of molecular oxygen, atmospheres  
 $q$  = Heat flux, BTU/ft<sup>2</sup>-sec  
 $q_{\text{conv}}$  = Convective heat flux with transpiration cooling, BTU/ft<sup>2</sup>-sec  
 $R$  = Universal gas constant, K cal/mole°K  
 $R_c$  = Fraction of carbon in material  
 $R_{O_x}$  = Ratio of oxygen to material oxidized  
 $T_{O_x}$  = Activation temperature of oxidation, °R  
 $T_s$  = Spalling temperature, °R  
 $T_w$  = Temperature at surface, °R  
 $X$  = Dimensionless oxidation rate  
 $Y$  = Dimensionless mass loss rate or oxidation parameter  
 $\delta$  = Boundary layer thickness, ft  
 $K$  = Transpiration coefficient, dimensionless

#### Subscripts

COLD - Cold wall conditions  
 HOT - Hot wall conditions  
 INIT - Initial trial value  
 e - Conditions outside boundary layer  
 R - Recovery conditions  
 w - Surface conditions  
 $\alpha$  - Species of ablating material

In the search for high temperature materials to withstand re-entry conditions, a considerable amount of attention has been given to the possible use of graphite and other carbonaceous materials. Because of its high temperature of sublimation, its superior strength to weight ratio, and its high emissivity, it would be an ideal material for a reradiating heat shield, but for one problem, that it oxidizes at a relatively rapid rate. Anti-oxidation coatings have been developed for graphite, but suitable coatings are generally restricted to temperatures of about 3200°F, little better than can be obtained with coated refractory metals. In principle, it would seem possible to coat graphite with a high temperature ceramic, such as zirconia or thoria, but such materials are too brittle for use as coatings. For these reasons, the oxidation of carbonaceous materials has been studied, and a number of tests have been made in a plasma arc facility to verify theoretical predictions, and to obtain fundamental properties.

It is generally agreed that the oxidation of carbonaceous materials can be divided into three regimes, depending upon the processes which determine the rate of oxidation. These are:

(1) The reaction rate control regime in which the oxidation rate is controlled by surface kinetics processes, including absorption of reactants on the surface, chemical reaction on the surface and desorption of products from the surface<sup>1</sup>.

(2) The diffusion control regime in which the oxidation rate is controlled by diffusion of reactants to the surface or diffusion of products from the surface<sup>1</sup>.

(3) The transition regime in which both diffusion and surface kinetics determine the oxidation rate.

The reaction rate control regime has been studied extensively, both theoretically and experimentally. Glasstone, et al<sup>1</sup> present relations derived from absolute reaction rate theory which give the reaction rate for a true surface reaction applicable to non-porous materials. Wheeler<sup>2</sup> has developed a model for the reaction of a gas in the pores of a solid which is applicable to a porous material. Blyholder and Eyring<sup>3&4</sup> further developed and obtained experimental confirmation for Wheeler's model.

TU, Davis, and Hottel<sup>5</sup> in 1934 and more recently, Moore and Zlotnick<sup>6</sup> have studied the transition regime. Moore and Zlotnick were quite sophisticated in their treatment of the boundary layer and surface kinetics, however, their study lacked the benefit of experimental confirmation. Bradshaw<sup>7</sup> has studied the transition regime experimentally, but it is not known whether his results in this regime have been compared to theory, quantitatively.

The diffusion control regime has received extensive theoretical study<sup>8-12</sup>. However, here again, there appears to be a lack of theoretical studies which have been confirmed by experimental data.

This paper is a study of all three oxidation regimes, and, in addition, of mass loss by spalling. Both theoretical and experimental techniques have been employed to establish techniques for the investigation of mass loss of carbonaceous materials in a gas stream. The methods developed should also prove invaluable for the investigation of carbonaceous materials incorporating additives, internally diffused coatings, etc., for the reduction of oxidation rates. They are not essentially limited to carbonaceous materials, although other materials have not been tested to date.

### 1.1 Summary

A proprietary method of forming reinforced carbonaceous materials of high strength and porosity was developed by Chance Vought Corp. A large number of plasma tests have been run on specimens of this material in the 180 KW plasma arc facility of the Ling-Temco-Vought Research Center. The cold wall heating rates for these tests ranged from 8 to 730 BTU/ft<sup>2</sup>-sec, as determined by calorimeters, the air enthalpies ranged from 3500 to 16,000 BTU/lb as determined by energy balance and sonic throat methods, and the surface temperatures ranged from 1650°F to 5600°F as determined by optical pyrometers with suitable corrections. These conditions are typical of those experienced by manned re-entry vehicles. A large number of these plasma arc tests were carried out by Carlson, et al<sup>13</sup>. In analyzing these results, it was noted that the surface recessions could be approximately correlated on the assumption that the rate of diffusion of oxygen to the surface could be predicted by what might be termed 'Lewis analogy'. A typical material studied was designated Matrix II. Several of the materials were intended to resist oxidation, and one appeared particularly successful. This was designated as Matrix III.

It was decided that the Lewis analogy might be used in predicting surface recession rates in IBM studies of ablators. To obtain correct predictions of surface recession at low temperatures, the oxidation reaction suggested by Nolan and Scala<sup>14</sup> was used in conjunction with the diffusion equation.

In order to verify the equations, the test results of Carlson, et al<sup>13</sup>, were reanalyzed. At the same time, equations were developed to predict the rate of mechanical erosion, or 'spalling', which was seen to occur at high temperatures, and methods were developed to obtain the material constants from test data. The results obtained for Matrices II and III are given in Section 3 of this paper.

A heat shield system is currently under development, in which a reinforced carbonaceous face is used (F. C. Smith<sup>15, 16</sup>). The results of a series of oxidation tests on this material are also given in Section 3.

## 1.2 Conclusions

1. A set of equations has been derived to account for surface recession rates due to oxidation and spalling, and a method has been developed to derive the required material constants from plasma arc test results. Using this approach, it has been shown that test results can be correlated to within experimental scatter. This report has been restricted to demonstrating the feasibility of the approach, and insufficient test data has been available to permit the application of statistical methods in obtaining correlation.

2. Materials tested to date are restricted to a particular formulation, referred to as reinforced carbonaceous material. It is believed, however, that the methods developed here are applicable to any material which undergoes oxidation, and in which the chemical form is relatively independent of temperature.

3. One particular material, referred to as Matrix III, showed particular promise as an oxidation resistant material. This relies on an internally diffused coating, as opposed to a surface coating, and is therefore not liable to suffer catastrophic failure.

4. Due to limitations imposed by the performance of the plasma arc, the effects of pressure could not be determined.

## 2. THEORY OF OXIDATION

There are three basic mechanisms by which the surface can recede on a material composed mainly of carbon or graphite, when exposed to a stream of high temperature air.

(1) Oxidation: This is further divided up into three regimes:

(a) The reaction controlled regime, at low temperatures, in which the air at the surface is oxygen rich, and oxidation is controlled by the chemical reaction rate.

(b) The transition regime, at intermediate temperatures, in which the rate of oxidation is sufficiently high to reduce the concentration of oxygen near the surface.

(c) The diffusion controlled regime, at high temperatures, in which the oxidation rate is controlled by the available supply of oxygen diffusing through the boundary layer to the surface.

(2) Spalling: This covers a multitude of poorly defined mechanisms by which material is removed from the surface in excess to that removed by oxidation. Spalling typically occurs at high temperatures, and is probably dependent on shear forces or total pressure.

(3) Sublimation: This is the fundamental ablation process, in which the material is converted to gas, and absorbs heat of vaporization in the process. The process of sublimation may be completely masked by spalling, so that it cannot be detected independently. Since sublimation of the surface of a heat shield is well understood, and since the temperatures considered in this study are below the sublimation temperature of carbon, it will not be considered here.

## 2.1 Derivation of Oxidation Equations

The rate of mass loss due to oxidation by molecular oxygen is given by the equation,

$$\dot{m}_E = k_{ox} (\rho_{O_2})_w^{1/2} e^{-T_{ox}/T_w} \quad 2.1.1$$

where  $\dot{m}_E$  is the effective mass loss per unit area due to oxidation, including material which does not oxidize, but is released in the oxidation process;  $k_{ox}$  is the reaction rate coefficient, sometimes referred to as the "specific reactivity";  $(\rho_{O_2})_w$  is the partial pressure of molecular oxygen at the surface;  $T_{ox}$  is the activation temperature, equal to  $E/R$ , where  $E$  is the activation energy; and  $T_w$  is the surface temperature. There is still some disagreement as to the precise form of this equation, which has been taken from Nolan and Scala (Reference 14). In particular, it is not certain that the index (order of reaction) should be 1/2. Blyholder and Eyring indicate that from 1510 to 1870°R for porous graphite the index is indeed 1/2. But for higher temperatures and/or non-porous graphite, they found values from zero to 3/4<sup>3,4</sup>. Furthermore, the specific reactivity is believed to be temperature dependent,<sup>1,3,4</sup>. However, Blyholder and Eyring indicate that for porous graphite the reactivity is proportional to temperature to the 1/4 power and that for non-porous graphite, the power is unity,<sup>3,4</sup>. Thus, the temperature dependence of  $K_{ox}$  is quite weak compared to the exponential term in Equation 1. Finally, the aforementioned authors found that above 1870°F for porous graphite  $T_{ox}$  changes.<sup>4</sup>

If atomic oxygen is present at the surface, a second term should be added to Eqn. 1. It has been assumed here that all oxygen reaching the surface is recombined.

The relationship between the rate of diffusion of oxygen and the rate of heat conduction through the boundary layer can be derived in terms of an effective Lewis number. We assume that the boundary layer thicknesses for heat conduction and diffusion are equal, and given by  $\delta$ . Then the heat transfer rate in the presence of transpiration cooling from the gaseous products of ablation,  $q_{slow}$  is (see also Section 2.3).

$$\begin{aligned} q_{slow} &= ([k/c_p] / \delta - \sum (\alpha) \dot{m}_\alpha K_\alpha) (i_R - i_w) \\ &= h_{EFF} (i_R - i_w) \end{aligned} \quad 2.1.2$$



where  $[\bar{k}/c_p]$  is a suitably averaged ratio of conductivity  $k$  divided by specific heat  $c_p$  in the boundary layer;  $\dot{M}_\alpha$  and  $K_\alpha$  are rates of injection and transpiration coefficients, respectively, for a given species  $\alpha$  in the gaseous products of ablation;  $i_R$  and  $i_w$  are the values of the enthalpy of the air at recovery and wall temperatures, respectively; and  $h_{EFF}$  is the effective skin coefficient.

The rate of diffusion of oxygen,  $\dot{M}_{Ox}$  per unit area, is given by;

$$\dot{M}_{Ox} = \bar{\rho} D (c_{Ox_e} - c_{Ox_w}) / \delta \quad 2.1.3$$

where  $\bar{\rho} D$  is a suitably averaged product of density  $\rho$ , and diffusion coefficient  $D$ , in the boundary layer; and  $c_{Ox_e}$ ,  $c_{Ox_w}$  are mass concentration ratios of oxygen at the outer surface of the boundary layer, and at the wall, respectively.

Noting that the Lewis number is defined by;

$$L = \bar{\rho} D c_p / k \quad 2.1.4$$

we can define an effective Lewis number,  $L_{EFF}$ , by;

$$\begin{aligned} L_{EFF} &= \frac{\bar{\rho} D}{([\bar{k}/c_p] - \delta \sum (\alpha) \dot{M}_\alpha K_\alpha)} \\ &= \frac{\dot{M}_{Ox}}{h_{EFF} (c_{Ox_e} - c_{Ox_w})} \end{aligned} \quad 2.1.5$$

after substituting from Eqns. 2 & 3.

The effective Lewis number includes the effects of transpiration cooling, which is assumed to block heat conduction and oxygen diffusion through the boundary layer in equal ratios. Since the Lewis number normally has a value close to unity, it may be assumed that  $L_{EFF}$  is also close to unity.

At sufficiently low temperatures, the oxygen reaching the surface is mainly molecular, and the partial pressure of oxygen near the wall is;

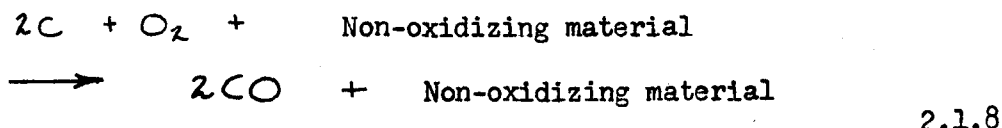
$$(p_{O_2})_w = \rho c_{Ox_w} (M.W. (AIR) / M.W. (O_2)) \quad 2.1.6$$

where  $\rho$  is the local static pressure; and  $M.W. (AIR)$ ,  $M.W. (O_2)$  are molecular weights of air and  $O_2$  respectively.

The ratio of oxygen consumed at the surface to the effective mass loss due to oxidation can be expressed as the constant ratio;

$$R_{ox} = \dot{m}_{ox} / \dot{m}_E \quad 2.1.7$$

if one chemical reaction is assumed to take place, for example, if the reaction is\*;



and if  $R_c$  is the proportion of carbon in the total material;

$$R_{ox} = (A.W.(ox)/A.W.(c)) R_c = \frac{4}{3} R_c \quad 2.1.9$$

where  $A.W.(ox)$  and  $A.W.(c)$  are atomic weights of oxygen and carbon, with values of 16 and 12 respectively.

Substituting the value of  $(p_{O_2})_w$  from Eqn. 2.1.6 into 2.1.1, and eliminating  $C_{oxw}$  and  $\dot{m}_{ox}$  from Eqns. 5 and 7 respectively.

$$\dot{m}_E = A p^{1/2} (1 - B \dot{m}_E / h_{EFF})^{1/2} e^{-T_{ox}/T_w} \quad 2.1.10$$

$$\text{where } A = k_{ox} \left\{ (M.W.(AIR) / M.W.(O_2)) C_{oxe} \right\}^{1/2} \quad 2.1.11$$

$$B = R_{ox} / L_{EFF} C_{oxe} \quad 2.1.12$$

Equation 2.1.10 gives the oxidation rate of carbon or graphite by molecular oxygen in the reaction rate control, transition, and diffusion rate control regimes. The terms  $A$ ,  $B$  and  $T_{ox}$  depend on the material and on the nature of the atmosphere. They are approximately constant for any given re-entry, provided that chemical reactions do not take place in the material which change the value of  $k_{ox}$ . The terms  $p$ ,  $h_{EFF}$ , and  $T_w$  define the environment at the surface, and vary during re-entry.

Additional material is lost by spalling and by sublimation. It is assumed that these can be characterized by an equation of the form;

$$\dot{m}_{SP} = C(p) e^{-T_s/T_w} \quad 2.1.13$$

where  $C(p)$  is a coefficient, dependent to an unknown extent on aerodynamic forces and other variables, and  $T_s$  is a characteristic spalling temperature, equivalent to the activation temperature  $T_{ox}$  in Eqn. 2.1.1.

The total mass loss rate now becomes;

$$\dot{m}_{TOT} = \dot{m}_E + \dot{m}_{SP} \quad 2.1.14$$

\* Blyholder and Eyring<sup>3,4</sup> indicate that CO is the primary product of graphite oxidation from 1510°R to 2230°R and probably to 2770°R.

## 2.2 Dimensionless Form of Equations

Equations 2.1.10 and 11 can be put into dimensionless form by taking

$$X = B \dot{m}_E / h_{EFF} = \text{dimensionless oxidation rate}$$

$$Y = B \dot{m}_{TOT} / h_{EFF} = \text{dimensionless mass loss rate or oxidation parameter}$$

and writing them in the form

$$X = K_0 (1 - X)^{1/2} e^{-T_{ox}/T_w} \quad 2.2.1$$

$$Y = X + K_s e^{-T_s/T_w} \quad 2.2.2$$

where

$$K_0 = AB \rho^{1/2} / h_{EFF} = \text{dimensionless reactivity}$$

$$K_s = BC / h_{EFF} = \text{dimensionless spalling constant}$$

The solution of Eqn. 1 is

$$X = \frac{1}{2} D \left\{ \sqrt{D^2 + 4} - D \right\} \quad 2.2.3$$

where

$$D = K_0 e^{-T_{ox}/T_w}$$

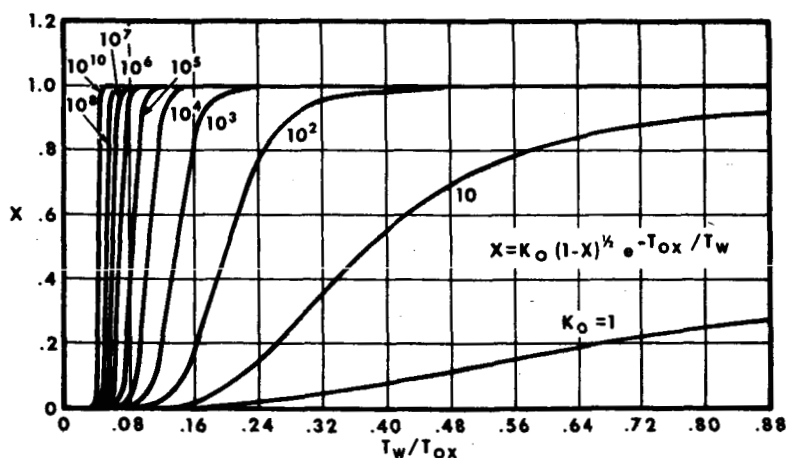


FIGURE 1 DIMENSIONLESS OXIDATION RATE (X) VS. TEMPERATURE RATIO  $T_w/T_{ox}$  FOR VARIOUS VALUES OF DIMENSIONLESS REACTION RATE  $K_0$

The dimensionless oxidation rate  $X$  is plotted in Figure 1 as a function of  $T_w/T_{ox}$  for a number of values of the dimensionless reactivity  $K_0$ . It is seen that at low temperatures the oxidation rate increases with temperature as governed by reaction kinetics, but as the temperature increases further, it undergoes transition to a constant rate determined by oxygen diffusion. Decreasing the reactivity has the effect of raising the

temperature at which transition occurs and of broadening the transition temperature regime. Furthermore, the extent to which decreasing the reactivity of a given type of material (by use of oxidation inhibitors for example) will decrease the oxidation rate depends strongly upon the temperature range of interest. It will be shown in Section 3 that the dimensionless form of the oxidation relations is extremely useful in evaluating experimental results.

### 2.3 Computation of Effective Skin Coefficient

Before plasma arc test results can be reduced, the effective skin coefficient  $h_{EFF}$  must be determined, as defined in Eqn. 2.1.2. Several methods are available for determining recovery enthalpy in the plasma stream,  $i_R$ , and the enthalpy at wall conditions,  $i_w$  can be determined from a Mollier diagram, if local pressure and surface temperature are known. The heat flux, however, is measured by a calorimeter having the same shape as the test specimen, and must be corrected for the effects of wall temperature and transpiration cooling.

The corrected convective heat flux can be written in the approximate form

$$q_{BLOW} = q_{HOT} - \sum (\alpha) \dot{M}_\alpha K_\alpha (i_R - i_w) \quad 2.3.1$$

where  $\dot{M}_\alpha$ ,  $K_\alpha$ ,  $i_R$ , and  $i_w$ , have already been defined in Eqn. 2.1.2, and  $q_{HOT}$  is the convective heating to the hot wall, which can be expressed in the form

$$q_{HOT} = q_{COLD} \left( \frac{i_R - i_w}{i_R - i_{COLD}} \right) \quad 2.3.2$$

Here,  $q_{COLD}$  is the equivalent heat flux to a cold wall, as measured by a calorimeter, where the enthalpy of the air at cold wall conditions is  $i_{COLD}$ .

### 2.4 Comparison with Method of Nolan and Scala

Nolan and Scala<sup>11</sup> use a somewhat different method for computing the oxidation rate, in which rates of oxidation in the reaction and diffusion regimes are first calculated, and then combined so that the result is asymptotically correct. Their approach can be summarized as follows:

The mass loss rate in the reaction controlled regime is given by Eqn. 2.1.1 in the form

$$\dot{M}_{REACT} = k_{ox} (p_{O_2})^{1/2} e^{-T_{ox}/T_w} \quad 2.4.1$$

The skin coefficient is computed from the equation

$$h_{EFF} = \frac{q_{HOT}}{i_R - i_w} = 0.0333 \frac{\rho^{1/2} f(\Lambda)}{\sqrt{2} R_\theta} \left[ \frac{\sqrt{2}}{f(\Lambda)} \right]^\delta + \text{TERM} \quad 2.4.2$$

where  $f(\Lambda)$  is a function of sweep angle,  $R_\theta$  is an effective nose radius,  $\delta$  is an index depending on geometry, and 'TERM' is a quantity which is small at high heating rates, and will be ignored in the following analysis. The mass loss rate in the diffusion controlled regime for a carbon or graphite material is given by

$$\dot{M}_{DIFF} = 6.2 \times 10^{-3} \frac{\rho^{1/2} f(\Lambda)}{\sqrt{2} R_\theta} \left[ \frac{\sqrt{2}}{f(\Lambda)} \right]^\delta \quad 2.4.3$$

Referring to Eqn. 2.1.10, it will be noted that the mass loss rate in the diffusion controlled regime can be obtained by equating the term in the brackets to zero, and solving for  $\dot{M}_E$ . If we then obtain  $h_{EFF}$  and  $\dot{M}_{DIFF}$  from Eqns. 2 and 3, with TERM = 0, we obtain

$$B = h_{EFF} / \dot{M}_{DIFF} = .0333 / 6.2 \times 10^{-3} = 5.37 \quad 2.4.4$$

This value is less than the 5.80 reported in Table II for carbon or graphite, as obtained from Eqn. 2.1.12. The mass loss rate in the transition regime is now approximated by

$$\dot{M}_E = \frac{1}{1/\dot{M}_{REACT} + 1/\dot{M}_{DIFF}} \quad 2.4.5$$

If we multiply this expression through by  $B/h_{EFF}$ , and substitute from Eqn. 4, noting that

$$\frac{B \dot{M}_{REACT}}{h_{EFF}} = D \quad 2.4.6$$

where  $D$  is defined in Eqn. 2.2.3, we have

$$X = \frac{1}{1/D + 1} \quad 2.4.7$$

This expression for  $D$  is compared with the expression derived in this report, as given by Eqn. 2.2.3, in Figure 2. It will be noted that the difference is not large.

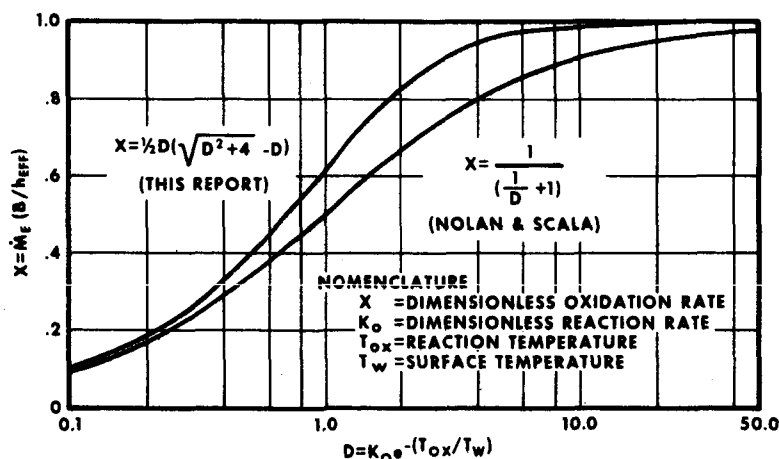


FIGURE 2 COMPARISON OF TWO EXPRESSIONS FOR THE DIMENSIONLESS OXIDATION RATE

### 3. EXPERIMENTAL RESULTS

#### 3.1 Determination of Oxidation Parameters from Plasma Tests

A large number of plasma tests have been run on specimens of reinforced carbonaceous material, or RCM, in the 180 kW plasma arc facility

of the Ling-Temco-Vought Research Center. The material referred to here is a proprietary system developed by Chance Vought Corp. It is described fully by Carlson, et al<sup>13</sup>, and consists of laminated sheets of carbon, or graphite cloth, bonded by a polymer, and then reduced to a char in an oven. The final result is a laminate bonded by long carbon molecules, possessing unusually good structural properties at high temperatures. Although the RCM is mainly carbon, its unique structure derives from its organic origin. Since the material is porous, it can be treated in a number of ways, including the vapor deposition of materials which improve its resistance to oxidation uniformly throughout its thickness. It can also be soaked in a 'filler' material, which ablates when heat is applied, thereby improving its effective insulative properties for limited periods. The RCM may also be graphitized, which modifies its structure somewhat.

Three materials were selected for a detailed study of their oxidation properties, and further information is given on their composition in Table I. A total of 81 tests is included in this study, and results are detailed in the Appendix.

TABLE I

Composition of Reinforced Carbonaceous Materials

Designation	Matrix II	Matrix III	Face Material
References	13	13	15, 16
Laminate	Graphite	Graphite	Graphite
Graphitized	No	Yes	No
Vapor Deposit	None	Silicon & Zirconium	None
Ablative Fillers	None Polystyrene Nylon 6 Fluoroalkyl Acrylate Ammonium Chloride Borox	None Nylon 6 Ammonium Chloride	None

The first two materials, Matrix II and Matrix III, were tested to determine their performance as ablators, as reported in Reference 13: Test specimens were cylindrical, 3/4 inches in diameter and 1/2 inches long, except for some specimens one inch long, and were tested under stagnation point conditions. The third material, referred to as face material, has been developed as a protective face for a special heat shield system; it

was tested under stagnation point conditions and also under side heating conditions, as reported in Reference 16. Configurations of these test specimens are shown in Figure 3.

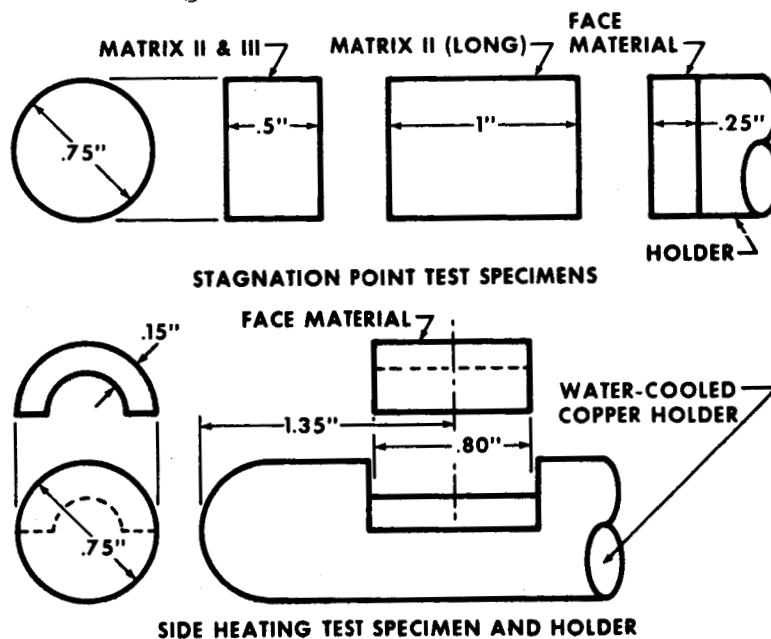


FIGURE 3 PLASMA ARC TEST SPECIMENS

### 3.2 Determination of Oxidation Parameters

The results of the plasma tests are given in the Appendix. Experimental values of the oxidation parameter  $\gamma$  are shown plotted in Figures 4, 5 and 6 for the material systems. In compiling the values of  $\gamma$ , it was necessary to assume a value for the effective Lewis number,  $(LEFF)_{INIT}$  so that  $\gamma$  is defined by

$$\gamma = \frac{B \dot{M}_{TOT}}{h_{EFF}} = \left( \frac{R_{OX} \dot{M}_{TOT}}{h_{EFF} C_{OXE}} \right) \frac{1}{(LEFF)_{INIT}} \quad 3.2.1$$

For any test point lying in the diffusion regime, and below the spalling regime, we should have

$$R_{OX} \dot{M}_{TOT} \approx R_{OX} \dot{M}_E = \dot{M}_{OX}$$

and

$$C_{OXW} \approx 0$$

Thus, from the definition of  $LEFF$  given by Eqn. 2.1.5

$$\gamma = LEFF / (LEFF)_{INIT} \quad 3.2.2$$

For the computations in the Appendix, and the experimental points plotted in Figures 4, 5 and 6, it was assumed that  $(LEFF)_{INIT} = 1$ . Thus, if a distinct diffusion regime exists, the values of  $\gamma$  for any experimental

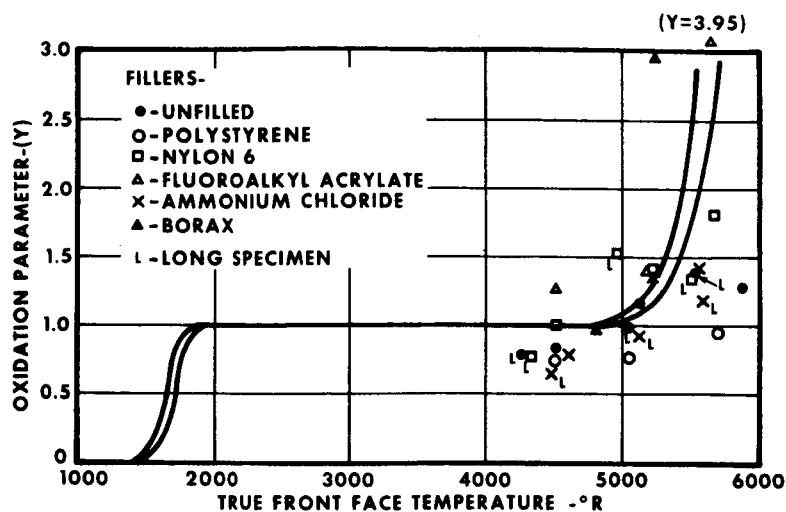


FIGURE 4 EXPERIMENTAL VS. THEORETICAL VALUES OF OXIDATION PARAMETER (Y) MATRIX II

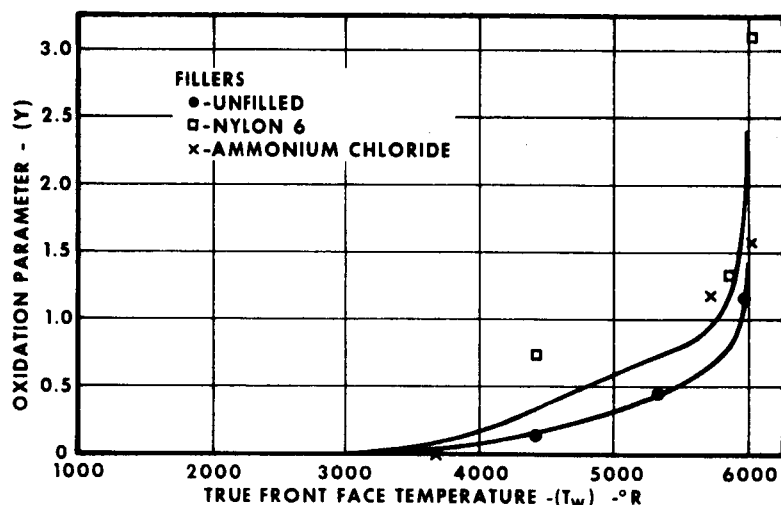


FIGURE 5 EXPERIMENTAL VS. THEORETICAL VALUES OF OXIDATION PARAMETER (Y) MATRIX III

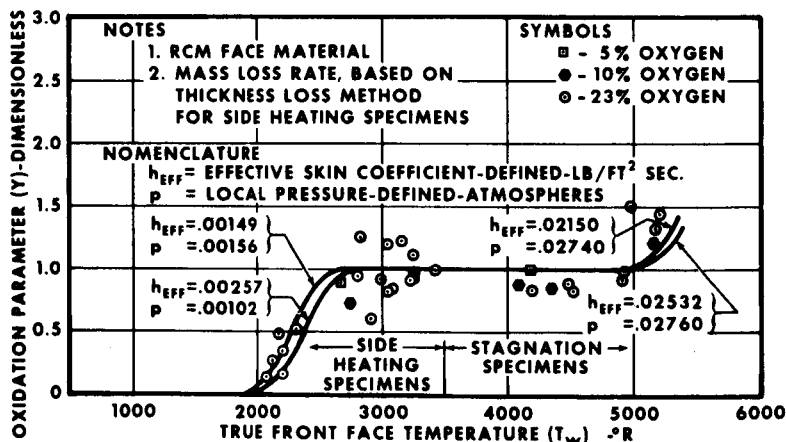


FIGURE 6 OXIDATION PARAMETER VS. SURFACE TEMPERATURE SIDE HEATING AND STAGNATION OXIDATION

points in this regime are equal to the experimental values of  $L_{EFF}$ . According to the theoretical curves shown in the figures (the derivation of these curves is explained later), distinct diffusion controlled regimes exist for Matrix II and face material. This regime is characterized by values of  $Y$  near unity and invariant with temperature. The number of experimental points for Matrix II, as shown in Figure 4, is insufficient to obtain a good value for  $L_{EFF}$  differing significantly from unity, although experimental points lie between 0.75 and 1.0. The experimental points for the face material, as shown in Figure 6, cover the complete diffusion control regime, and values for  $L_{EFF}$  lie between 0.8 and 1.25.

For lack of more precise information, it has been assumed that  $L_{EFF} = 1.0$ . From kinetic theory estimates<sup>17</sup> the Lewis number is of the order of one. Since the rate of loss of carbonaceous material, without oxidation protection, is very nearly proportional to  $L_{EFF}$  during re-entry trajectory, a better definition of



$L_{EFF}$  is evidently required. It may be noted that the results of Nolan and Scala<sup>11</sup> indicate that  $B = 5.37$  for carbon and graphite (see Eqn. 2.4.4), whereas  $B = 5.8$  when  $L_{EFF} = 1$  (see Table II). Thus Nolan and Scala's analytical results indicate a value for  $L_{EFF}$  of  $5.8/5.37 = 1.08$ , when applied to a typical nose cone, in comparison with the experimental range of 0.75 to 1.25 obtained in plasma tests. In an earlier paper<sup>9</sup> Scala assumed  $L_{EFF} = 1.2$ . Most other investigators<sup>8, 10, 12</sup> have assumed  $L_{EFF} = 1.0$ .

Experimental values for  $L_{EFF}$  presented here reflect the assumption that transpiration affects diffusion as well as convective heat transfer through the boundary layer. For specimens without filler, the transpiration cooling reduced the heat flux by less than 11% in all cases, and had it been ignored, experimental values of  $L_{EFF}$  would have been reduced by the same percentages. However, for specimens with ablative filler, reductions lay between 16% and 49%, which would have brought the corresponding experimental points well below those for the unfilled specimens. Although the evidence is not conclusive, it does appear necessary to consider transpiration effects when computing oxidation rates in the presence of ablation. When there is no ablation, it does not have much effect on the results, whether or not it is correct.

The experimental results presented in the Appendix have been used to determine values of the oxidation and spalling constants, by methods explained in the following two sections. The results are given in Table II, and have been used to compute the theoretical curves in Figures 4, 5 and 6. Because of the range of values of  $p$  and  $h_{EFF}$  during the tests, two theoretical curves are shown in each figure, representing the range of experimental conditions. These are summarized in Table III. For comparison, values of the oxidation constants are included in Table II for commercial and pyrolytic graphite, as given by Nolan and Scala<sup>11, 18</sup> and Horton<sup>19</sup>.

The test results for Matrix II, as shown in Figure 4, lie mainly in the spalling regime characterized by values of  $\gamma$  considerably greater than unity, and show considerable scatter about the theoretical curve. More regularity can be discerned in the test points for any given material (matrix-filler composite), indicating that the filler material may have an effect on spalling.

The test results for Matrix III, as shown in Figure 4, indicate considerably better agreement with the theoretical curves. In fact, the only bad point is one with nylon filler. The writers have viewed motion pictures of a plasma test on material incorporating nylon, and it was evident that the nylon extrudes from the surface in plastic form. It is possible that this is responsible for mechanical erosion of the surface, causing an excessive mass loss. The significant point about the tests on the Matrix III material was that it exhibited considerable resistance to oxidation. This is evidenced by values of  $\gamma$  in the reaction rate and transition control regimes ( $\gamma$  considerably less than unity) up to extremely high temperatures. In fact the mass loss rate did not exceed the theoretical diffusion controlled rate ( $\gamma = 1.0$ ) until a surface temperature of 5750°R was reached, where spalling began to occur.

TABLE II

## SUMMARY OF PROPERTIES

MATERIAL	MATRIX II	MATRIX III	FACE	COMMERCIAL PYROLYTIC GRAPHITE	GRAPHITE (ALTERNATE)	PYROLYTIC GRAPHITE (ALTERNATE)
References				14, 18	14, 18	14, 19
Density, lb/ft <sup>3</sup>	62.4	62.4	62.4	108	138	138
R <sub>c</sub>	1.0	.865-.925	1.0	1.0	1.0	1.0
Act. Energy, K cal/mole	(1) 44	36.6	44	44	44	47.5
T <sub>ox</sub> , °R	(1) 39,800	32,400	39,800	39,800	39,800	29,700
R <sub>ox</sub> , lb/ft <sup>2</sup> SEC ATM <sup>1/2</sup>	(1) 6.73 x 10 <sup>8</sup>	20.2	3.76 x 10 <sup>5</sup>	6.73 x 10 <sup>8</sup>	4.47 x 10 <sup>4</sup>	3.07 x 10 <sup>4</sup>
(2) A, lb/ft <sup>2</sup> SEC ATM <sup>1/2</sup>	3.06 x 10 <sup>8</sup>	9.23	1.72 x 10 <sup>5</sup>	3.08 x 10 <sup>8</sup>	2.05 x 10 <sup>4</sup>	1.41 x 10 <sup>4</sup>
(2) B	5.8	5.03-5.36	5.8			0.59 x 10 <sup>4</sup>
T <sub>s</sub> , °R	157,500	346,600	(3) 157,500			
Mean P for spalling, atm	.07	.07				
C, lb/ft <sup>2</sup> SEC	1.36 x 10 <sup>10</sup>	6.88 x 10 <sup>22</sup>	(3) 1.36 x 10 <sup>10</sup>			

Assumed the same as commercial graphite

Values for 23% oxygen

Assumed same as Matrix II

TABLE III  
CONSTANTS FOR THEORETICAL OXIDATION PARAMETERS  
(23% Oxygen)

MATERIAL		MATRIX II	MATRIX III	FACE
$T_{ox}$ , °R		39,800	32,400	39,800
$A$ , lb/ft <sup>2</sup> SEC ATM <sup>1/2</sup>		$3.06 \times 10^8$	9.23	$1.72 \times 10^5$
$B$		5.8	5.22	5.8
$T_s$ , °R		157,500	346,500	157,500
$C$ , lb/ft <sup>2</sup> SEC		$1.36 \times 10^{10}$	$6.88 \times 10^{22}$	$1.36 \times 10^{10}$
Transition Regime:				
$p$ , atmospheres,	Max	0.065	0.065	.00156
	Min	0.05	0.05	.00102
$h_{EFF}$ , lb/ft <sup>2</sup> SEC	Max	0.04	0.04	.002570
	Min	0.02	0.02	.001491
$K_0$	Max	$2.28 \times 10^{10}$	615	$2.63 \times 10^7$
	Min	$10^{10}$	270	$1.245 \times 10^7$
Spalling Regime:				
$p$ , atmospheres,	Max	0.065	0.065	.02760
	Min	0.05	0.05	.02740
$h_{EFF}$ , lb/ft <sup>2</sup> SEC.	Max	0.04	0.04	.02532
	Min	0.02	0.02	.02150
$K_s$	Max	$3.95 \times 10^{12}$	$18 \times 10^{24}$	$3.66 \times 10^{12}$
	Min	$1.97 \times 10^{12}$	$9 \times 10^{24}$	$3.11 \times 10^{12}$

The plasma arc tests reported on Matrices II and III were originally carried out to determine the effectiveness of RCM as an ablative material. Although the test results were used in developing the theory presented here, the presence of fillers introduced added complications. The tests on the face material were carried out specifically to determine its oxidation properties, and both side heating and stagnation point tests were made to increase the range of surface temperatures. The results shown in Figure 6 cover tests in 5, 10 and 23% oxygen, mixed with nitrogen, and fall into the transition, diffusion, and spalling regimes. Scatter of the test points about the theoretical curves is highest for the side heating specimens in the transition regime, but this may be due to experimental difficulties. Mass losses had to be kept small because the specimens were only 0.15 inches thick, and erosion rates varied considerably over the surfaces, due to flow effects. The mass losses actually given were computed from recessions measured at the same locations as the calorimeters used in determining the cold wall heat fluxes. Mass losses at the stagnation point were obtained by weighing, however, mass losses were also computed from the measured recession of the face material specimen (in brackets in the Appendix) to determine whether oxidation occurred in depth. Comparison of the two sets of figures indicates that this is not the case.

Points corresponding to 0% oxygen could not be included in this plot. Therefore, the mass loss rates,  $\dot{M}_{TOT}$ , are shown in Figure 7 for all tests on the face material. Three of the stagnation point specimens in 0% oxygen showed considerable mass losses at around 3,500°R, while the side heating specimens at lower temperatures showed no mass loss. Since the losses occurred well below the spalling regime as evidenced by the results in Figure 6, no reasonable explanation can be given.

Due to the severe restrictions on the range of pressures obtainable in the plasma arc, it was not possible to check the dependency of oxidation and spalling on pressure. Other methods are available to verify the half order of reaction assumed in Eqn. 2.1.1, but the determination of the pressure dependency of the spalling coefficient (C in Eqn. 2.1.13) will present a serious problem.

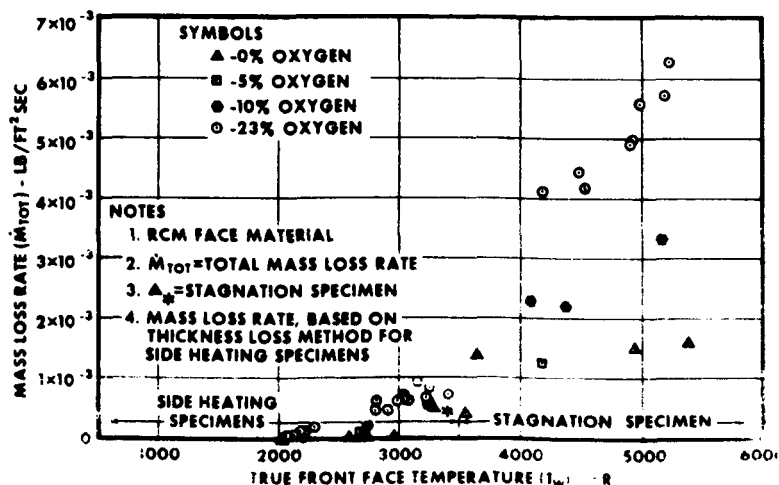


FIGURE 7 MASS LOSS RATE VS. SURFACE TEMPERATURE  
SIDE HEATING AND STAGNATION OXIDATION

## 3.3

Determination of Oxidation Constants

Experimental values of the oxidation parameter  $Y$  obtained in the transition regime, and at temperatures for which spalling is negligible, can be used to determine the oxidation constants. In the temperature range defined, we have  $Y=X < \lambda$ , and can rewrite Eqn. 2.2.1 in the form

$$P_{ox} = \frac{Y^2}{1-Y} \frac{h_{EFF}}{P} = (AB)^2 e^{-2T_{ox}/T_w} \quad 3.3.1$$

Taking logarithms of both sides

$$\ln P_{ox} = \ln (AB)^2 - 2 T_{ox}/T_w \quad 3.3.2$$

Thus if experimental values of  $P_{ox}$  are plotted against  $1/T_w$  on semilog paper, the slope of the best fit line is equal to  $-2 T_{ox} \log_{10} e$  and the intercept is equal to  $\log_{10} (AB)^2$ . A plot of this type is shown in Figure 8 for Matrix III material, and includes all test points below  $5500^\circ R$ . Another plot is shown in Figure 9 for the face material, and includes six side heating test points below  $2300^\circ R$ . The test point for nylon filler is questionable, as explained in Section 3.2. All of these tests were made in 23% oxygen. The values of  $P_{ox}$  shown in the two figures were obtained from the Appendix.

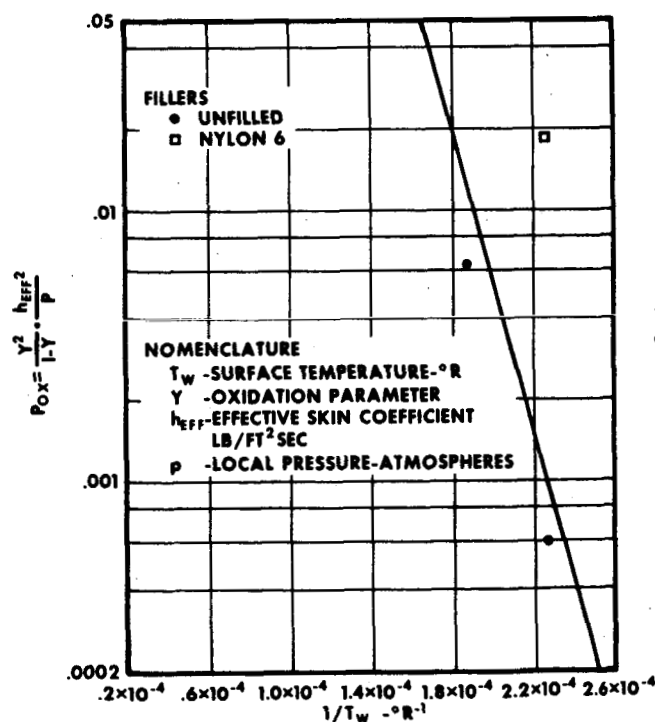


FIGURE 8 GRAPHICAL DETERMINATION OF OXIDATION CONSTANTS MATRIX III

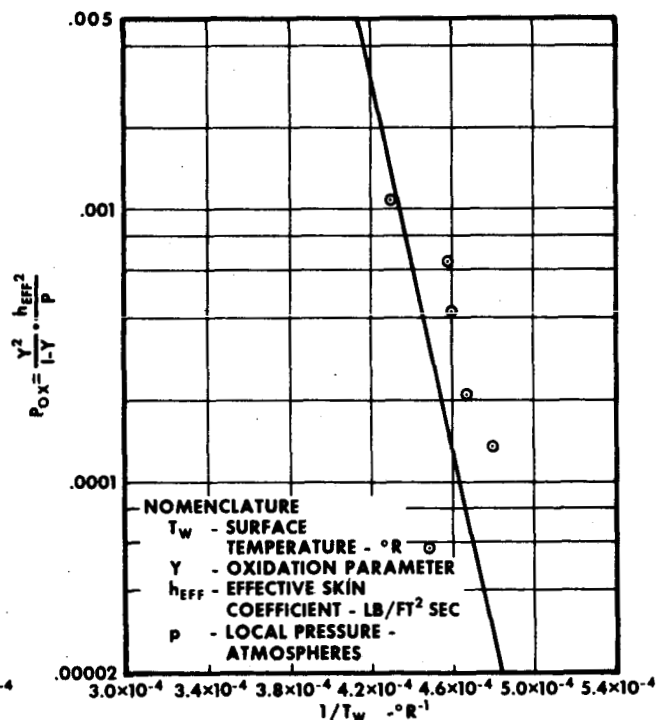


FIGURE 9 GRAPHICAL DETERMINATION OF OXIDATION CONSTANTS RCM FACE MATERIAL

Values of  $A$  and  $T_{ox}$  obtained by estimated best fit lines in Figures 8 and 9 are recorded in Table II. For a check on the validity of these results, the lower portions of the theoretical oxidation parameter curves in Figures 5 and 6 may be referred to. No tests on Matrix II material were made at sufficiently low temperatures to get into the transition regime, so that oxidation constants could not be determined. The values used for the theoretical curves in Figure 4 were based on the values given for commercial graphite in Table II.

Although it is always possible to find a pair of values of  $A$  and  $T_{ox}$  by this method, which will fit the test data fairly closely on the oxidation parameter curves, there is insufficient data to determine either one value accurately. Thus, a value for  $A$  (or  $k_o$ ), given independently of  $T_{ox}$ , does not define the oxidation performance adequately.

### 3.4 Determination of Spalling Constants

Experimental values of the oxidation parameter  $Y$  obtained in the spalling regime, can be used to determine the spalling constants, once the oxidation constants have been determined. First, we solve for  $X$  in Eqn. 2.2.1, using values of  $A$ ,  $B$ , and  $T_{ox}$ , as given in Table II. We then re-write Eqn. 2.2.2 in the form

$$P_{SPALL} = (Y - X) h_{EFF} = BC e^{-T_s/T_w} \quad 3.4.1$$

Taking logarithms of both sides

$$\ln P_{SPALL} = \ln(BC) - T_s/T_w \quad 3.4.2$$

Thus if experimental values of  $P_{SPALL}$  are plotted against  $1/T_w$  on semilog paper, the slope of the best fit line is equal to  $-T_s \log_{10} e$ , and the intercept is equal to  $\log_{10}(BC)$ . Plots of this type are shown for Matrices II and III respectively in Figures 10 and 11. The values of  $P_{SPALL}$  were taken from Table IV.

Values of  $C$  and  $T_s$  obtained in this way are recorded in Table II, and were used for the theoretical plots of the oxidation parameter in Figures 4 and 5. Insufficient high temperature tests were carried out on the face material to determine its spalling constants, and the theoretical curves in Figure 6 assume the same values as for Matrix II.

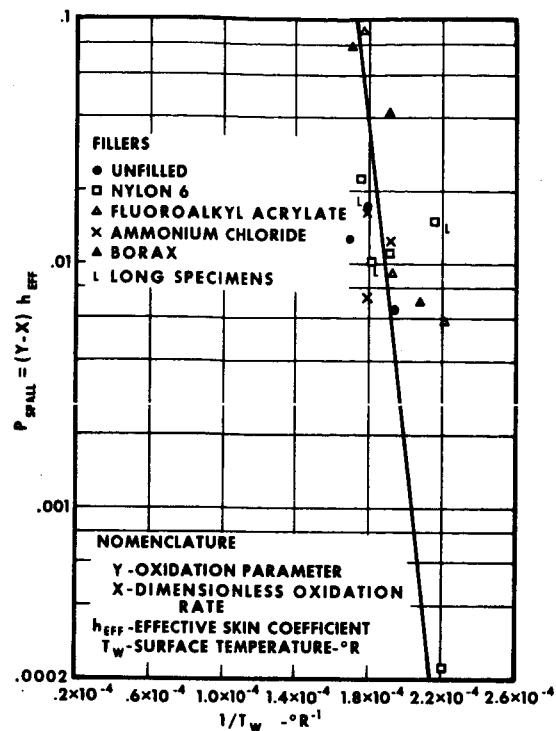


FIGURE 10 GRAPHICAL DETERMINATION OF SPALLING CONSTANTS MATRIX II

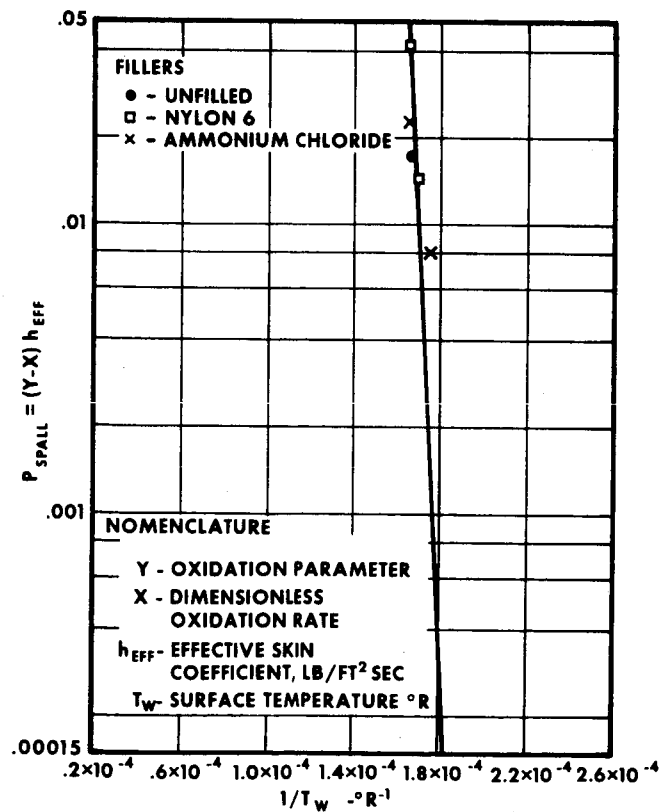


FIGURE 11 GRAPHICAL DETERMINATION OF SPALLING CONSTANTS MATRIX III

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## APPENDIX

### Definition of Terms in Plasma Test Data Summary

Type:	Stagn. or Side for stagnation point or side heating test, respectively.
Specimen No:	Number used to denote specimen in test series. The Matrix II specimens with capital letter, e.g., II-A-1, are long specimens.
Material:	Designation of carbonaceous material and filler as defined in Table I.
A	Area of specimen (ft <sup>2</sup> ).
R <sub>ox</sub>	Ratio of oxygen to carbonaceous material (= 1.33 R <sub>c</sub> , where R <sub>c</sub> is proportion of carbon, as given in Table see Eqns. 2.1.7, 8 and 9).
ΔM <sub>M</sub>	Total mass loss of carbonaceous material (lb). By direct measurement on stagnation point specimens. Computed from local recession on side heating specimens. Mass losses computed from recession on the stagnation point face specimens are shown in brackets.
ΔM <sub>A</sub>	Total mass loss of ablative filler (lb). Specimens were run until back face temperature indicated loss of filler.
t	Duration of heat pulse in plasma arc test (secs).
C <sub>oxe</sub>	Mass fraction of oxygen in plasma stream.
B	Diffusion parameter; = $R_{ox} / C_{oxe} (L_{EFF})_{INIT}$
i <sub>a</sub>	Enthalpy of plasma stream (BTU/lb).
p	Local static pressure (atmospheres). Stagnation pressure behind shock assumed for stagnation point specimens. Free stream pressure assumed for side heating specimens.
q <sub>cold</sub>	Cold wall heating rate to calorimeter mounted at stagnation point for stagnation point specimens. Mounted on side of sting for side heating specimens. Reading taken immediately before and after each test run (BTU/ft <sup>2</sup> -sec).
T <sub>w</sub>	Stabilized surface temperature of specimen (°R). Taken by optical pyrometer, and corrected for emissivity and viewing window.
$\dot{M}_{TOT}$	Mass loss rate of carbonaceous material (lb/ft <sup>2</sup> -sec). = $\Delta M_M / tA$

$\dot{M}_A$	Mass loss rate of ablative filler (lb/ft <sup>2</sup> -sec). $= \Delta M_A / t_A$
$i_w$	Enthalpy of test medium at wall conditions (BTU/lb), i.e., temperature $T_w$ and pressure $p$ .
$\Delta i$	Driving enthalpy (BTU/lb) $= i_R - i_w$
$q_{HOT}$	Hot wall heat flux (BTU/ft <sup>2</sup> -sec) $= q_{COLD} \Delta i / (i_R - i_{COLD})$ , ( $i_{COLD} = 210-260$ BTU/lb. for stagnation specimens and 130 BTU/lb. for side heating specimens).
$\dot{M}_E'$	Mass loss rate in diffusion regime (lb/ft <sup>2</sup> -sec). $= (q_{HOT} / \Delta i - K \dot{M}_A) / (B + K)$ This is a solution of the eqns. $B \dot{M}_E' / h_{EFF} = 1$ $h_{EFF} = q_{HOT} / \Delta i - K (\dot{M}_E' + \dot{M}_A)$
$\dot{M}_E''$	Lesser of $\dot{M}_E'$ and $\dot{M}_{TOT}$ (lb/ft <sup>2</sup> -sec). This is the maximum amount of carbonaceous material which can have oxidized on the surface, and in the boundary layer after spalling, and can therefore have contributed to transpiration cooling.
$q_{BLOW}$	Convective heating rate corrected for transpiration cooling (BTU/ft <sup>2</sup> -sec). $= q_{HOT} - K \Delta i (\dot{M}_E'' + \dot{M}_A)$ ( $K$ assumed equal to .67 for all cases).
$h_{EFF}$	Effective skin coefficient (lb/ft <sup>2</sup> -sec). $= q_{BLOW} / \Delta i$
$Y$	Oxidation parameter $= B \dot{M}_{TOT} / h_{EFF}$
$P_{OX}$	Quantity used in determining oxidation constants (see Eqn. 3.3.1). $= \frac{Y^2}{1-Y} \cdot \frac{h_{EFF}^2}{\rho}$
$X$	Dimensionless oxidation rate (see Eqn. 2.2.3). $= \frac{1}{2} D \left\{ \sqrt{D^2 + 4} - D \right\}$ where $D = \frac{A B \rho^{1/2} e^{-T_{OX}/T_w}}{h_{EFF}}$
	For Matrix II and face material, $X \approx 1$ in the spalling regime. For Matrix III material, $A$ and $T_{OX}$ are taken from Table II.
$P_{SPALL}$	Quantity used in determining spalling constants (see Eqn. 3.4.1). $= (Y - X) h_{EFF}$

# PLASMA TEST DATA SUMMARY

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	II-a-1	II-a-2	II-a-3	II-b-1	II-b-2	II-b-3
Material	Matrix II	Matrix II	Matrix II	Matrix II Polystyrene	Matrix II Polystyrene	Matrix II Polystyrene
A	.00304	.00304	.00305	.00304	.00306	.00304
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.001980	.001630	.000990	.001930	.000510	.000620
ΔM <sub>A</sub>	0	0	0	.003200	.003150	.003240
t	115.0	67.0	33.0	142.0	48.5	42.0
C <sub>ox</sub>	.23	.23	.23	.23	.23	.23
B	5.80	5.80	5.80	5.80	5.80	5.80
i <sub>R</sub>	5350	9650	14300	5350	9500	14600
P	.05068	.06245	.07007	.05068	.06122	.07075
q <sub>COLD</sub>	226	418	692	226	398	719
T <sub>w</sub>	4520	5140	5880	4510	5050	5700
M <sub>TOT</sub>	.005660	.007980	.009830	.004470	.003440	.004860
M <sub>A</sub>	0	0	0	.007410	.021220	.025380
i <sub>w</sub>	1400	1880	2750	1400	1800	2550
Δi	3950	7770	11550	3950	7700	12050
q <sub>HOT</sub>	173	345	569	173	330	603
M <sub>E</sub>	.006770	.006860	.007610	.006000	.004430	.005110
M <sub>E</sub>	.005660	.006960	.007610	.004470	.003440	.004860
q <sub>BLOW</sub>	158.0	309.3	510.1	141.6	202.9	358.9
h <sub>EFF</sub>	.040010	.039810	.044200	.035800	.026300	.029800
Y	.8208	1.1628	1.2898	.7243	.7586	.9460
P <sub>ox</sub>	---	---	---	---	---	---
X	---	1.0	1.0	---	---	---
P <sub>SPALL</sub>	---	.00648	.01281	---	---	---

# PLASMA TEST DATA SUMMARY (Cont'd)

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	II-c-1	II-c-2	II-c-3	II-d-1	II-d-2	II-d-3
Material	Matrix II Nylon 6	Matrix II Nylon 6	Matrix II Nylon 6	Matrix II Fluor Acr	Matrix II Fluor Acr	Matrix II Fluor Acr
A	.00304	.00305	.00305	.00305	.00305	.00304
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000990	.000970	.001020	.000700	.000830	.002600
ΔMA	.002980	.002830	.002910	.004060	.004310	.002580
t	64.0	48.5	39.0	48.5	50.0	40.0
Cox <sub>e</sub>	.23	.23	.23	.23	.23	.23
B	5.80	5.80	5.80	5.80	5.80	5.80
i <sub>R</sub>	5400	9500	14600	5400	9500	14600
P	.05102	.06122	.07075	.05102	.06122	.07075
q <sub>COLO</sub>	224	398	674	221	406	707
T <sub>w</sub>	4520	5230	5680	4520	5190	5640
M <sub>TOT</sub>	.005090	.006560	.008580	.004730	.005440	.021380
M <sub>A</sub>	.015320	.019130	.024460	.027450	.028260	.021220
L <sub>w</sub>	1400	2000	2520	1450	1940	2500
Δi	4000	7500	12080	3950	7560	12100
q <sub>HOT</sub>	172	322	567	168	332	596
M <sub>E</sub>	.005060	.004650	.004720	.003730	.003860	.005420
M <sub>E</sub>	.005060	.004650	.004720	.003730	.003860	.005420
q <sub>BLOW</sub>	117.4	202.5	330.8	85.5	169.3	380.0
h <sub>EFF</sub>	.029300	.027000	.027400	.021640	.022400	.031400
Y	1.0075	1.4093	1.8161	1.2676	1.4085	3.9490
P <sub>ox</sub>	---	---	---	---	---	---
X	1.0	1.0	1.0	1.0	1.0	1.0
P <sub>SMALL</sub>	.00022	.01105	.02236	.00579	.00915	.09260

PLASMA TEST DATA SUMMARY (Cont'd)

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	II-e-1	II-e-2	II-e-3	II-g-1	II-g-2	II-g-3
Material	Matrix II Amm Chl	Matrix II Amm Chl	Matrix II Amm Chl	Matrix II Borax	Matrix II Borax	Matrix II Borax
A	.00304	.00304	.00305	.00305	.00305	.00305
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000900	.000940	.001410	.003420	.003300	.004940
ΔM <sub>A</sub>	.001510	.001450	.001440	.003240	.003500	.002910
t	63.0	40.0	50.0	168.0	98.0	94.0
c <sub>oxe</sub>	.23	.23	.23	.23	.23	.23
B	5.80	5.80	5.80	5.80	5.80	5.80
i <sub>R</sub>	5500	9500	14500	5600	9750	14500
p	.05136	.06122	.07041	.05170	.06218	.07080
q <sub>COLD</sub>	230	412	684	214	316	458
T <sub>w</sub>	4610	5220	5560	4810	5240	5840
M <sub>TOT</sub>	.004700	.007730	.009250	.006670	.011040	.017230
M <sub>A</sub>	.007880	.011920	.009440	.006320	.011710	.010150
i <sub>w</sub>	1450	1960	2400	1600	2000	2700
Δi	4050	7540	12100	4000	7750	11800
q <sub>HOT</sub>	176	335	580	158	258	380
M <sub>E</sub>	.005900	.005790	.006430	.005450	.005650	.006360
M <sub>E</sub>	.004700	.005790	.006430	.005450	.005650	.006360
q <sub>BLOW</sub>	141.9	245.5	451.3	126.5	167.9	249.5
h <sub>EFF</sub>	.035000	.032600	.037300	.031600	.021700	.021100
Y	.7789	1.3752	1.4383	1.2244	2.9507	4.7360
P <sub>ox</sub>	---	---	---	---	---	---
X	---	1.0	1.0	1.0	1.0	1.0
P <sub>SPALL</sub>	---	.01223	.01635	.00709	.04233	.07883

PLASMA TEST DATA SET 1871 (Cont'd)

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	II-A-1	II-A-2	II-A-3	II-C-1	II-C-2	II-C-3
Material	Matrix II	Matrix II	Matrix II	Matrix II Nylon 6	Matrix II Nylon 6	Matrix II Nylon 6
A	.00308	.00307	.00306	.00307	.00307	.00307
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.001300	.001520	.002200	.001910	.002170	.001910
ΔM <sub>A</sub>	0	0	0	.005380	.004940	.005100
t	244.0	61.0	69.0	135.0	95.0	90.0
Cox <sub>e</sub>	.23	.23	.23	.23	.23	.23
B	5.80	5.80	5.80	5.80	5.80	5.80
i <sub>R</sub>	5400	9300	11400	5400	9300	11000
P	.05068	.06122	.07075	.05068	.06109	.07007
q <sub>COLD</sub>	242	482	682	239	502	730
T <sub>w</sub>	4260	5060	5540	4340	4960	5510
M <sub>TOT</sub>	.005770	.008120	.010400	.004600	.007430	.006930
M <sub>A</sub>	0	0	0	.012980	.016900	.019600
i <sub>w</sub>	1260	1800	2350	1280	1960	2320
Δi	4140	7500	12050	4120	7540	11980
q <sub>HOT</sub>	194	398	580	189	335	622
M <sub>HE</sub>	.007240	.008200	.007440	.005740	.005120	.006000
M <sub>E</sub>	.005770	.008120	.007440	.004600	.005120	.006000
q <sub>BLOW</sub>	178.0	358.8	519.9	143.2	210.8	361.5
h <sub>EFF</sub>	.043000	.047800	.043100	.034800	.028100	.029930
Y	.7784	.9854	1.3995	.7667	1.5335	1.3428
P <sub>ox</sub>	---	---	---	---	---	---
X	---	---	1.0	---	1.0	1.0
P <sub>SMALL</sub>	---	---	.01722	---	.01499	.01026

PLASMA TEST DATA SUMMARY (Cont'd)

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	II-E-1	II-E-2	II-E-3	III-a-1	III-a-2	III-a-3
Material	Matrix II Annul Chl	Matrix II Annul Chl	Matrix II Annul Chl	Matrix III	Matrix III	Matrix III
A	.00308	.00308	.00308	.00296	.00296	.00296
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.2226	1.1560	1.2000
ΔM <sub>M</sub>	.001770	.001770	.002360	.000060	.000140	.000430
ΔM <sub>A</sub>	.002860	.002500	.002920	0	0	0
t	131.0	82.0	94.0	22.7	13.0	16.0
C <sub>oxe</sub>	.23	.23	.23	.23	.23	.23
B	5.80	5.80	5.80	5.32	5.03	5.22
i <sub>R</sub>	5400	9300	14300	3500	8000	13000
P	.05068	.06122	.07007	.05170	.06333	.07177
q <sub>COLD</sub>	242	500	724	144	326	590
T <sub>w</sub>	4490	5130	5590	4420	5340	5960
M <sub>TOT</sub>	.001390	.007010	.008150	.000950	.003540	.009000
M <sub>A</sub>	.007090	.009900	.010090	0	0	0
i <sub>w</sub>	1380	1870	2420	1330	2100	2950
Δi	4020	7430	11880	2170	5900	10150
q <sub>HOT</sub>	187	409	612	94	248	470
M <sub>E</sub>	.006460	.007480	.006920	.007230	.007370	.007860
M <sub>E</sub>	.001390	.007010	.006920	.000950	.003540	.007860
q <sub>GLOW</sub>	157.9	324.8	476.6	92.6	234.0	416.6
h <sub>EFF</sub>	.039280	.043720	.040120	.042680	.039660	.041040
Y	.6482	.9300	1.1782	.1291	.4491	1.1447
P <sub>ox</sub>	---	---	---	.000597	.006275	---
X	---	---	1.0	.15276	.49843	.72578
P <sub>SMALL</sub>	---	---	.00715	---	---	.01719



PLASMA TEST DATA SUMMARY (Cont'd)

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	III-c-1	III-c-2	III-c-3	III-e-1	III-e-2	III-e-3
Material	Matrix III Nylon 6	Matrix III Nylon 6	Matrix III Nylon 6	Matrix III Amm Chl	Matrix III Amm Chl	Matrix III Amm Chl
A	.00296	.00296	.00296	.00290	.00289	.00290
R <sub>ox</sub>	1.2000	1.2266	1.2320	1.1933	1.1813	1.1813
ΔM <sub>M</sub>	.000790	.000670	.001150	0	.000420	.000690
ΔM <sub>A</sub>	.003040	.003500	.003450	.001140	.001640	.001040
t	66.1	42.5	33.6	39.0	29.0	25.3
c <sub>ox</sub>	.23	.23	.23	.23	.23	.23
B	5.22	5.33	5.36	5.19	5.14	5.14
i <sub>R</sub>	3500	10350	13000	3500	9800	13000
p	.05136	.06293	.07143	.05116	.06190	.07143
q <sub>COLD</sub>	138	365	572	129	374	563
T <sub>w</sub>	4420	5860	6040	3680	5720	6020
M <sub>TOT</sub>	.004040	.005330	.011520	0	.005010	.009400
M <sub>A</sub>	.015540	.027820	.034570	.010080	.019570	.011170
i <sub>w</sub>	1330	2730	2920	1000	2600	2900
Δi	2170	7620	10080	2500	7200	10100
q <sub>HOT</sub>	91	275	452	97	282	446
M <sub>A</sub>	.005350	.002910	.003600	.005470	.004490	.005970
M <sub>E</sub>	.004040	.002910	.003600	0	.004490	.005970
q <sub>slow</sub>	62.5	118.1	194.2	80.1	155.9	309.7
h <sub>EFF</sub>	.028820	.015500	.019270	.032050	.021660	.030660
Y	.7318	? 1.8329	3.1028	0	1.1888	1.5760
P <sub>ox</sub>	.018650	? ---	---	---	---	---
X	.21800	? .91149	.91541	---	.81736	.82103
P <sub>small</sub>	.01480	? .01428	.04215	---	.00805	.02315

# PLASMA TEST DATA SUMMARY (Cont'd)

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	P1	P2	P3	P4	P5	P6
Material	Face	Face	Face	Face	Face	Face
A	.00304	.00305	.00305	.00304	.00301	.00304
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.001060 [.001030]	.000940 [.000830]	.000390 [.000860]	.000940 [.000950]	.001180 [.001190]	.001040 [.001080]
ΔM <sub>A</sub>	0	0	0	0	0	0
t	69.5	75.0	73.0	70.0	70.0	54.5
C <sub>ox</sub>	.23	.23	.23	.23	.23	.23
B	5.80	5.80	5.80	5.80	5.80	5.80
i <sub>R</sub>	10800	6700	6850	9000	15900	16633
P	.02640	.02300	.02320	.02510	.02740	.02820
q <sub>COLD</sub>	343	202	210	280	395	480
T <sub>w</sub>	4933	4195	4539	4488	4990	5224
M <sub>TOT</sub>	.005017	.004109	.004176	.004417	.005600	.006277
M <sub>A</sub>	0	0	0	0	0	0
i <sub>w</sub>	1830	1250	1440	1570	1870	1760
Δi	8970	5450	5410	7430	14030	14873
q <sub>HOT</sub>	296	170	172	237	354	436
M <sub>E</sub>	.005103	.004833	.004910	.004940	.003903	.004531
M <sub>E</sub>	.005017	.004109	.004176	.004417	.003903	.004531
q <sub>BLOW</sub>	266.0	155.4	156.7	215.5	317.6	390.9
h <sub>EFF</sub>	.022656	.028517	.028970	.022004	.022640	.026281
Y	.9812	.8357	.3861	.8833	1.4346	1.3853
P <sub>ox</sub>	---	---	---	---	---	---
X	---	---	---	---	---	---
P <sub>SPALL</sub>	---	---	---	---	---	---

# PLASMA TEST DATA SUMMARY (Cont'd)

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	P <sub>7</sub>	P <sub>8</sub>	P <sub>9</sub>	P <sub>10</sub>	P <sub>11</sub>	P <sub>12</sub>
Material	Face	Face	Face	Face	Face	Face
A	.00304	.00302	.00304	.00304	.00305	.00304
R <sub>OX</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000580	.000210	.000430	.000370	.000970	.001060
	[.000679]	[.000160]	[.000460]	[.000250]	[.000980]	[.000860]
ΔM <sub>A</sub>	0	0	0	0	0	0
t	133.3	161.0	93.0	283.0	55.5	280.6
COX <sub>g</sub>	0	0	0	0	.23	.05
B	∞	∞	∞	∞	5.80	26.67
i <sub>R</sub>	8708	3590	10175	3391	15108	6358
P	.02260	.01650	.02320	.01590	.02760	.01930
q <sub>COLD</sub>	439	155	460	139	433	206
T <sub>w</sub>	3664	3312	4958	3569	5190	4194
M <sub>TOT</sub>	.001379	.000493	.001520	.000430	.005730	.001242
M <sub>A</sub>	0	0	0	0	0	0
L <sub>w</sub>	1000	880	1860	960	2140	1250
Δi	7608	2710	8315	2431	12968	4118
q <sub>HOT</sub>	400	124	386	105	378	139
M <sub>H</sub>	0	0	0	0	.004507	.001232
M <sub>E</sub>	0	0	0	0	.004507	.001232
q <sub>BLOW</sub>	---	---	---	---	339.0	135.3
h <sub>EFF</sub>	---	---	---	---	.026143	.033234
Y	---	---	---	---	1.2712	1.0085
P <sub>OX</sub>	---	---	---	---	---	---
X	---	---	---	---	---	---
P <sub>SMALL</sub>	---	---	---	---	---	---

PLASMA TEST DATA SUMMARY (Cont'd)

Type	Stagn.	Stagn.	Stagn.	Stagn.	Stagn.
Specimen No.	P <sub>13</sub>	P <sub>11</sub>	P <sub>15</sub>	P <sub>16</sub>	P <sub>17</sub>
Material	Face	Face	Face	Face	Face
A	.00305	.00305	.00304	.00304	.00304
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000250	.001430	.000900	.000970	.001560
	[.000210]	[.001280]	[.000940]	[.000830]	[.001600]
ΔM <sub>A</sub>	0	0	0	0	0
t	36.0	141.0	184.0	145.0	104.0
Cox <sub>e</sub>	.10	.10	.0	.10	.23
B	13.33	13.33	∞	13.33	5.80
i <sub>R</sub>	6100	12817	11258	6517	12270
P	.02140	.02570	.02080	.02170	.02640
q <sub>COLD</sub>	211	483	538	223	402
T <sub>w</sub>	4100	5182	5398	4380	4920
M <sub>TOT</sub>	.002276	.003325	.001608	.002200	.004934
M <sub>A</sub>	0	0	0	0	0
i <sub>w</sub>	1200	2140	2470	1360	1880
Δi	14900	10677	8788	5157	10390
q <sub>HOT</sub>	176	407	430	183	348
M <sub>E</sub>	.002572	.002722	0	.002538	.005173
M <sub>E</sub>	.002276	.002722	0	.002200	.004934
q <sub>BLOW</sub>	169.0	387.3	---	165.6	313.4
h <sub>EFF</sub>	.034482	.036276	---	.032114	.030166
Y	.8799	1.2218	---	.9132	.9487
P <sub>ox</sub>	---	---	---	---	---
X	---	---	---	---	---
P <sub>SPALL</sub>	---	---	---	---	---

PLASMA TEST DATA SUMMARY (Cont'd)

Type	Side	Side	Side	Side	Side	Side
Specimen No.	SH-1	SH-2	SH-4	SH-5	SH-6	SH-7
Material	Face	Face	Face	Face	Face	Face
A	.00640	.00640	.00642	.00656	.00656	.00665
R <sub>OX</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000036	.000359	.000093	.000500	.000457	.000024
ΔM <sub>A</sub>	0	0	0	0	0	0
t	121.0	120.0	121.0	120.0	119.5	488.0
c <sub>OXE</sub>	.23	.23	.23	.23	.23	0
B	5.80	5.80	5.80	5.80	5.80	∞
i <sub>R</sub>	5130	10600	5500	13500	13450	4724
p	.00156	.00293	.00177	.00333	.00333	.00068
q <sub>COLD</sub>	8.50	32.70	10.65	49.00	53.50	9.18
T <sub>W</sub>	2223	2822	2218	3263	3270	2040
M <sub>TOT</sub>	.000047	.000467	.000119	.000635	.000583	.000007
M <sub>A</sub>	0	0	0	0	0	0
i <sub>W</sub>	570	740	570	870	880	520
Δi	4560	9860	4930	12630	12570	4204
q <sub>HOT</sub>	7.77	30.82	9.80	46.32	50.50	8.43
M <sub>E</sub>	.000263	.000483	.000307	.000567	.000621	0
M <sub>E</sub>	.000047	.000467	.000119	.000567	.000583	0
q <sub>BLOW</sub>	7.63	27.99	9.44	41.52	45.62	---
h <sub>EFF</sub>	.001673	.002839	.001915	.003287	.003629	---
Y	.1632	.9542	.3603	1.1205	.9317	---
P <sub>OX</sub>	.000057 ?	.054690	.000421	---	---	---
X	---	---	---	---	---	---
P <sub>SMALL</sub>	---	---	---	---	---	---

PLASMA TEST DATA SUMMARY (Cont'd)

Type	Side	Side	Side	Side	Side	Side
Specimen No.	SH-8	SH-9	SH-10	SH-11	SH-12	SH-13
Material	Face	Face	Face	Face	Face	Face
A	.00657	.00660	.00653	.00663	.00662	.00661
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000070	.000075	.000362	0	.000655	.000218
ΔM <sub>A</sub>	0	0	0	0	0	0
t	480.0	428.0	75.0	71.0	150.0	70.0
c <sub>oxe</sub>	.0	0	.23	.23	.23	.23
B	∞	∞	5.80	5.80	5.80	5.80
i <sub>R</sub>	10835	17095	14300	6650	12800	10000
p	.00153	.00255	.00306	.00119	.00255	.00323
q <sub>COLO</sub>	30.30	51.80	68.04	11.80	58.32	41.00
T <sub>w</sub>	2506	2974	3436	2220	3234	2926
M <sub>TOT</sub>	.000022	.000026	.000739	0	.000660	.000471
M <sub>A</sub>	0	0	0	0	0	0
i <sub>w</sub>	670	780	950	560	860	770
Δi	10165	16315	13350	6090	11940	9230
q <sub>HOT</sub>	28.80	49.84	64.15	11.04	55.00	38.38
M <sub>E</sub>	0	0	.000743	---	.001712	.0006343
M <sub>E</sub>	0	0	.000739	---	.000660	.000471
q <sub>BLOW</sub>	---	---	57.54	---	49.72	35.47
h <sub>EFF</sub>	---	---	.004310	---	.004164	.003843
Y	---	---	.9944	---	.9193	.7106
P <sub>ox</sub>	---	---	---	---	---	---
X	---	---	---	---	---	---
P <sub>SPALL</sub>	---	---	---	---	---	---

PLASMA TEST DATA SUMMARY (Cont'd)

Type	Side	Side	Side	Side	Side	Side
Specimen No.	SH-14	SH-15	SH-16	SH-17	SH-18	SH-19
Material	Face	Face	Face	Face	Face	Face
A	.00654	.00653	.00659	.00656	.00660	.00656
R <sub>OX</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000435	.000199	0	.000097	.000617	.000650
ΔM <sub>A</sub>	0	0	0	0	0	0
t	99.5	961.0	468.0	480.0	960.0	480.0
c <sub>OXE</sub>	.23	0	0	0	.05	.10
B	5.80	∞	∞	∞	26.67	13.33
i <sub>R</sub>	11100	8331	7110	10691	9758	7608
p	.00323	.00153	.00153	.00255	.00170	.00204
q <sub>COLO</sub>	55.60	41.44	17.00	57.10	28.48	28.51
T <sub>w</sub>	3061	2717	2165	2744	2690	2750
M <sub>TOT</sub>	.000662	.0000032	0	.000031	.000097	.000206
M <sub>A</sub>	0	0	0	0	0	0
i <sub>w</sub>	320	720	550	720	700	720
Δi	10280	7611	6560	9971	9058	6888
q <sub>HOT</sub>	52.15	38.51	16.00	53.96	26.82	26.30
M <sub>E</sub>	.000784	0	0	0	.000108	.000273
M <sub>E</sub>	.000662	0	0	0	.000097	.000206
q <sub>BLOW</sub>	47.59	---	---	---	26.23	25.35
h <sub>EFF</sub>	.004629	---	---	---	.002896	.003680
Y	.9296	---	---	---	.8933	.7462
P <sub>OX</sub>	---	---	---	---	.036900	.014390
X	---	---	---	---	---	---
P <sub>SMALL</sub>	---	---	---	---	---	---

PLASMA TEST DATA SUMMARY (Cont'd)

Type	Side	Side	Side	Side	Side	Side
Specimen No.	SH-20	Spare 2	Spare 3	SH-1R	SH-2R	SH-10R
Material	Face	Face	Face	Face	Face	Face
A	.00656	.00657	.00667	.00650	.00618	.00626
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000095	.001170	.001350	.000191	.000902	.000931
ΔM <sub>A</sub>	0	0	0	0	0	0
t	242	235	240	240	240	240
c <sub>oxe</sub>	.23	.23	.23	.23	.23	.23
B	5.80	5.80	5.80	5.80	5.80	5.80
i <sub>R</sub>	3350	12400	10600	5400	11000	13000
p	.00102	.00255	.00323	.00156	.00279	.00340
q <sub>COLD</sub>	9.67	60.75	57.78	8.30	33.30	59.40
T <sub>w</sub>	2085	3177	3277	2195	2832	3116
M <sub>TOT</sub>	.000060	.000938	.000845	.000124	.000608	.000619
M <sub>A</sub>	0	0	0	0	0	0
i <sub>w</sub>	540	850	880	560	740	830
Δi	3310	11550	9720	4340	10260	12170
q <sub>HOT</sub>	8.63	57.23	53.69	7.62	31.90	56.17
M <sub>E</sub>	.000403	.000766	.000854	.000243	.000481	.000713
M <sub>E</sub>	.000060	.000766	.000854	.000124	.000481	.000619
q <sub>BLOW</sub>	8.50	51.30	48.19	7.22	28.60	51.12
h <sub>EFF</sub>	.002570	.004442	.004958	.001491	.002788	.004200
Y	.1353	1.2247	.9885	.4822	1.2647	.8548
P <sub>ox</sub>	.000137	---	---	.063990	---	---
X	---	---	---	---	---	---
P <sub>SMALL</sub>	---	---	---	---	---	---



# PLASMA TEST DATA SUMMARY (Cont'd)

Type	Side	Side	Side	Side
Specimen No.	SH-11R	SH-13R	SH-14R	SH-20R
Material	Face	Face	Face	Face
A	.00668	.00644	.00633	.00649
R <sub>ox</sub>	1.3333	1.3333	1.3333	1.3333
ΔM <sub>M</sub>	.000296	.001092	.000962	.000131
ΔM <sub>A</sub>	0	0	0	0
t	240	240	240	240
C <sub>oxe</sub>	.23	.23	.23	.23
B	5.80	5.80	5.80	5.80
i <sub>R</sub>	5800	10000	12000	5600
p	.00224	.00361	.00374	.00157
q <sub>COLD</sub>	12.15	37.50	52.11	9.72
T <sub>w</sub>	2319	3067	3011	2011
M <sub>TOT</sub>	.000184	.000706	.000633	.000084
M <sub>A</sub>	0	0	0	0
i <sub>w</sub>	600	320	790	550
Δi	5050	11210	9180	5200
q <sub>HOT</sub>	11.14	34.88	49.21	9.08
M <sub>E</sub>	.000331	.000587	.000679	.000273
M <sub>E</sub>	.000184	.000587	.000633	.000084
q <sub>BLOW</sub>	10.50	31.27	44.46	8.80
h <sub>EFF</sub>	.002019	.003406	.003966	.001743
Y	.5285	1.2023	.9256	.2794
P <sub>ox</sub>	.001078	---	.048430	.000211
X	---	---	---	---
P <sub>SMALL</sub>	---	---	---	---